FCC QUALITY RULES

(Applicable to contracts concluded on or after 01 March 2008)

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QUALITY RULES

APPLICABLE TO CONTRACTS CONCLUDED ON OR AFTER 01 MARCH 2008

PART 1: GENERAL

1. APPLICATION OF QUALITY RULES

The following Quality Rules apply to all contracts incorporating the FCC Contract Rules for Cocoa Beans.

2. DEFINITIONS

In addition to the definitions in the FCC Contract Rules for Cocoa Beans, the following definitions are applicable to the FCC Quality Rules.

2.1 ADULTERATION

Means alteration of the composition of a parcel of cocoa beans by any means whatsoever so that the resulting mixture or combination does not conform to the contractual description.

2.2 BEAN CLUSTER

Means two or more beans joined together which cannot be separated by finger and thumb.

2.3 BEAN COUNT

Means the total number of whole beans per 100 g derived from a test sample prepared in accordance with these Rules.

2.4 BROKEN BEAN

Means a cocoa bean of which a fragment is missing, the remaining part being more than half of a whole bean.

2.5 COCOA BEAN

Means a raw cocoa bean, which is the whole seed of the cocoa tree (Theobroma Cacao L.)

2.6 CONTAMINATION

Means cocoa which has a smoky, hammy or other off-flavour taste or smell, or which contains a substance not natural to cocoa.

2.7 DEFECTIVE BEAN

Means an internally mouldy or insect-damaged bean.

2.8 FAIR AVERAGE QUALITY

Means the quality specification for that season applicable to the cocoa origin referred to in the contract when the terms Good Fermented and Fair Fermented are not customarily applicable to that origin.

2.9 FAIR FERMENTED
Means cocoa beans that are not more than 10% slaty and 10% defective by count.

2.10 FLAT BEAN
Means a cocoa bean which is too thin to be cut to give a complete surface of the cotyledons.

2.11 FOREIGN MATTER
Means any substance other than cocoa beans and residue.

2.12 FRAGMENT
Means a piece of cocoa bean equal to or less than half a bean.

2.13 GERMINATED BEAN
Means a cocoa bean the shell of which has been pierced, split or broken by the growth of the seed-germ.

2.14 GOOD FERMENTED
Means cocoa beans that are not more than 5% slaty and 5% defective by count.

2.15 INSECT DAMAGED/INFESTED BEAN
Means a cocoa bean the internal parts of which are found to contain insects or mites at any stage of development, or to show signs of damage caused thereby, which are visible to the naked eye.

2.16 MOULDY BEAN
Means a cocoa bean on the internal parts of which mould is visible to the naked eye.

2.17 RESIDUE
Means any cocoa element other than whole cocoa beans and flat beans which does not pass through the sieve (eg. broken beans, fragments and pieces of shell).

2.18 SIEVE
Means a screen with round holes the diameter of which shall be 5.0mm min./max.

2.19 SIEVINGS
Means the material which will pass through the Sieve.

2.20 SLATY BEAN
Means a cocoa bean which shows a slaty colour on half or more of the exposed surface of the cotyledons by the cut test.

3. SPECIFICATION

3.1 GENERAL

Parties shall specify their quality requirements e.g. Main Crop, Good Fermented, Fair Fermented, the Bean Count, in the FCC Short Form Contract. Parties may also agree to incorporate the Optional Quality Clauses set out in Part 3.

In addition to any specified quality terms, the parcel shall consist of beans which shall be reasonably;

- uniform in size,
- uniform in fermentation,
- dry,
- homogeneous in all other respects

and the parcel shall be:

- fit for the production of a foodstuff,
- virtually free from foreign matter and adulteration, contamination, live insects (including mites), rodents or other type of infestation,
- reasonably free from bean clusters, flat beans, germinated beans, residue and sievings in accordance with Rule 3.3.

3.2 BEAN SIZE STANDARDS

Bean size is defined by the bean count and is usually expressed by the number of beans per 100g.
For Main Crop, the following shall apply:

(a) if the bean count is 100 or less, then the parcel shall not be subject to an allowance;
(b) if the bean count is between 101 and 120 inclusive, the parcel shall be subject to an allowance;
(c) if the bean count exceeds 120, then the parcel shall be replaceable or subject to an allowance.

3.3 SIEVINGS STANDARDS

If the weight of the Sievings exceeds 1.5% of the weight of the whole arbitration sample the arbitrators may award an allowance.

3.4 EXCESSIVE RESIDUE, FLAT BEANS, BEAN CLUSTERS AND FOREIGN MATTER

If the residue and/or flat beans and/or bean clusters and/or foreign matter in the arbitration sample are considered excessive the arbitrators may award an allowance.

4. QUALITY ASSESSMENT

The quality of the parcel will be assessed in accordance with the following procedure:

1. A sample shall be drawn in accordance with the FCC Sampling Rules.
2. The Assessment of Sievings shall be conducted according to the methodology set out in Rule 5.
3. The Assessment of Bean Count shall be conducted according to the methodology set out in Rule 6.
4. The Assessment of Slaty and Defective Beans shall be conducted by a Cut Test according to the methodology set out in Rule 7.

Quality tests for the Optional Quality Clauses must be conducted in accordance with the relevant methodology set out in Part 4 of these Quality Rules.

PART 2: STANDARD METHODOLOGIES

5. METHODOLOGY FOR ASSESSING THE SIEVINGS

5.1 PREPARATION OF THE SAMPLE

The Arbitration Sample shall be prepared in accordance with the method described in the FCC Sampling Rules.

5.2 DETERMINATION

The whole of the Arbitration Sample shall be weighed, well mixed and then sieved through a screen with round holes the diameter of which shall be 5.0mm min./max.

The quantity passing through the sieve, which is known as the Sievings, shall be collected and weighed.

The percentage of the Sievings is obtained by comparing the weight of the Sievings against the total net weight of the Arbitration Sample.

5.3 EXPRESSION OF RESULT

$$\text{Sievings (\%)} = \frac{\text{Weight of the sievings}}{\text{Total net weight of sample}} \times 100$$

6. METHODOLOGY FOR ASSESSING THE BEAN COUNT

6.1 PRINCIPLE

The Bean count is to determine the average number of whole cocoa beans that weigh 100g.

6.2 PREPARATION OF THE ARBITRATION SAMPLE

The Arbitration Sample shall be prepared in accordance with the FCC Sampling Rules.

6.3 PREPARATION OF THE TEST SAMPLE

After sieving in accordance with Rule 5, the Arbitration Sample must be emptied onto a clean dry flat surface and thoroughly mixed.

A Test Sample of not less than 600 grammes shall be obtained by using a flat-bottomed shovel drawn across the middle of an Arbitration Sample and weighed to the nearest 1 g.

6.4 DETERMINATION

From the Test Sample the residue, foreign matter, flat beans and any bean clusters shall be removed and then weighed and replaced by an equivalent weight of whole beans taken randomly from the remainder of the Arbitration Sample. The total number of beans in the Test Sample shall then be counted and the resulting number is the Bean Count and shall be expressed by the number of beans per 100 grammes.

6.5 EXPRESSION OF RESULT

The bean count shall be expressed as number of beans per 100 g.

$$\text{Thus, bean count} = \frac{\text{Number of whole beans}}{\text{Weight of whole beans (g)}} \times 100$$

7. METHODOLOGY FOR THE CUT TEST

7.1 PRINCIPLE

The cut-test is to determine the incidence of beans which are defective and/or germinated and/or slaty.

7.2 PREPARATION OF THE TEST SAMPLE

The Test Sample of whole beans used for the Bean Count test shall be the sample used for the Cut Test procedure as set out in this Rule.

7.3 DETERMINATION

Three hundred whole beans irrespective of size, shape and condition, shall be counted off from the Test Sample and the beans shall be cut lengthwise through the middle, such that the maximum cut surface of the cotyledons can be examined by the naked eye.

Both halves of each bean shall be examined visually in full daylight or equivalent artificial light.

Separate counts shall be made of the number of beans which are defective and/or slaty. Where a bean is defective in more than one respect, only one defect shall be counted.

7.4 EXPRESSION OF RESULT

The results for defect shall be expressed as a percentage of the number of beans examined.

PART 3: OPTIONAL QUALITY CLAUSES

The methodologies of the International Confectionery Association ("the ICA methodologies") shall always be subject to the provisions of the FCC Rules as defined in Rule 1.2 of the FCC Contract Rules for Cocoa Beans. In the event of a conflict between the ICA methodologies and the FCC Rules then the FCC Rules shall take precedence unless the contrary is expressly provided for.

8. FREE FATTY ACIDS (FFA)

8.1 DEFINITION

The free fatty acid content is the percentage by mass of the free fatty acids in the cocoa fat, conventionally expressed as oleic acid (molecular weight 282).

8.2 PREPARATION AND ANALYSIS OF THE SAMPLE

At least one arbitration sample shall be prepared in accordance with the FCC Sampling Rules. The Buyer shall instruct his supervisor/agent to forward the arbitration sample to an agreed independent analyst. The arbitration sample shall be analysed in accordance with Rule 8.5. The costs of the analysis shall be paid by the Buyer.

8.3 ARBITRATION CLAIMS

Arbitration claims for FFA shall be supported by an original certificate of analysis issued by an independent analyst in accordance with Rule 8.2.

8.4 TIME LIMITS

The time limits specified in the FCC Contract Rule for Cocoa Beans 20.1.1shall apply for FFA claims.

8.5 FREE FATTY ACID ANALYSIS

The free fatty acid content shall be determined in accordance with Analytical Method 42 (1993) of the International Confectionery Association as set out in Part 4 except that Rule 7 of Analytical Method 42 shall not apply and sampling shall instead be carried out pursuant to Rule 8.2 of these FCC Quality Rules.

8.6 STANDARDS

The Buyer and Seller shall state in the contract the percentage by mass of the free fatty acids in the cocoa fat below which a claim will not be pursued ("the permitted percentage") and a maximum level above which the parcel shall be replaceable ("the maximum percentage"). For values between the permitted percentage and the maximum percentage or where Buyer and Seller have not agreed a maximum percentage, the parcel shall be subject to an allowance.

8.7 APPROVED WORDING FOR SPECIFIC CONTRACTS

Free Fatty Acids, Optional Quality Rule 8, shall apply to this contract.
The Buyer may not claim for excessive free fatty acid if the percentage by mass of free fatty acids in the cocoa fat shall be "xxx" or less.

The Buyer may claim an allowance if the percentage by mass of free fatty acids in the cocoa fat shall be greater than "xxx" but less than "yyy".

The Buyer may claim for replacement of the parcel if the percentage by mass of free fatty acids in the cocoa fat shall be “yyy” or greater.

The name and address of the independent analyst upon which the parties have agreed:
Name:
Address:

9. MOISTURE CONTENT OF COCOA BEANS

9.1 DEFINITION

The moisture content of cocoa beans is conventionally the loss in mass of beans, determined by the International Confectionery Association (ICA) Analytical Method 43 - 1993 and expressed in % m/m.

9.2 PREPARATION AND ANALYSIS OF THE SAMPLE

At least one arbitration sample shall be prepared in accordance with the FCC Sampling Rules. The arbitration sample shall be placed in an air-tight container which must be filled completely by the sample to ensure that the moisture level remains constant. This container shall also be sealed by the Buyer's sampler and, if appointed, the Seller's Superintendent. The Buyer's sampler shall immediately forward the arbitration sample to an independent analyst to be mutually agreed by both the Buyer and Seller at the time of the contract in respect of which neither shall unreasonably withhold its consent. The arbitration sample will be analysed in accordance with Rule 9.5.The costs of the analysis shall be paid by the Buyer.

9.3 ARBITRATION CLAIMS

Arbitration claims for excessive moisture shall be supported by an original certificate of analysis issued by an independent analyst in accordance with Rule 9.2.

9.4 TIME LIMITS

The time limits specified in the FCC Contract Rules for Cocoa Beans 20.1.1 shall apply for claims concerning moisture content.

9.5 MOISTURE CONTENT ANALYSIS

The moisture content shall be determined in accordance with Analytical Method 43 (1993) of the International Confectionery Association as set out in Part 4 except that Rule 6 of Analytical Method 43 shall not apply and sampling shall instead be carried out pursuant to Rule 9.2 of these FCC Quality Rules.

9.6 STANDARDS

The Buyer and Seller shall state in the contract the percentage by mass of moisture in the cocoa beans below which a claim for excessive moisture will not be pursued ("the permitted percentage") and, if required, a maximum level above which the parcel shall be replaceable ("the maximum percentage").

For values between the permitted percentage and the maximum percentage or where Buyer and Seller have not agreed a maximum percentage, the parcel shall be subject to an allowance.

9.7 APPROVED WORDING FOR SPECIFIC CONTRACTS

Moisture content, Optional Quality Rule 9, shall apply to this contract.

The Buyer may not claim for excessive moisture content if the percentage by mass of moisture in the cocoa beans shall be "xxx" or less.

The Buyer may claim an allowance if the percentage by mass of moisture in the cocoa beans shall be greater than "xxx" but less than "yyy".

The Buyer may claim for replacement of the parcel if the percentage by mass of moisture in the cocoa beans shall be "yyy" or greater.

The name and address of the independent analyst upon which the parties have agreed:
Name:
Address:

10. OFF-FLAVOURS IN COCOA BEANS

10.1 DEFINITION

Off-flavours in cocoa beans for the purpose of this Rule means smoke off-flavour, mould off-flavour, hammy off-flavour and acidic off-flavour as defined in and determined by analysis according to Analytical Method 44 – 1996 of the International Confectionery Association (ICA), of a sample taken from a particular parcel.

10.2 PREPARATION AND ANALYSIS OF THE SAMPLE

At least one arbitration sample shall be prepared in accordance with the FCC Sampling Rules. Should the Buyer consider that an off-flavour be present, the Buyer's sampler shall forward the arbitration sample to an independent taste-panel to have been mutually agreed by both the Buyer and Seller at the time of the contract in respect of which neither shall unreasonably withhold its consent. The costs of the analysis shall be paid by the Buyer.

10.3 ARBITRATION CLAIMS

Arbitration claims for off-flavours shall be supported by an original certificate of analysis issued by an independent analyst in accordance with Rule 10.2.

10.4 TIME LIMITS

The time limits specified in the FCC Contract Rules for Cocoa Beans 20.1.1 shall apply for claims for off-flavours.

10.5 OFF-FLAVOUR ANALYSIS

The off-flavour analysis shall be determined according to Analytical Method 44 – 1996 of the International Confectionery Association as set out in Part 4.

10.6 APPROVED WORDING FOR SPECIFIC CONTRACTS

Off-flavours, Optional Quality Rule 10, shall apply to this contract.

The name and address of the independent taste-panel upon which the parties have agreed:
Name:
Address:

PART 4: OPTIONAL QUALITY CLAUSES METHODOLOGIES

11. METHODOLOGY FOR DETERMINATION OF FREE FATTY ACID (FFA)

Analytical Method 42

Determination of the Free Fatty Acid (FFA) Content of Cocoa Fat as a Measure of Cocoa Nib Acidity

Introduction

This method describes the determination of the free fatty acid content in fat obtained by soxhlet extraction of cocoa nib with petroleum ether.

1. Scope and Field of Application

Applicable to cocoa nib prior to processing.

2. References

BS 684: Section 2.10: 1988, Determination of acidity.

3. Definition

The free fatty acid content (acidity) is the percentage by mass of free fatty acids in cocoa fat, conventionally expressed as oleic acid (molecular weight 282), determined according to the method described below.

4. Principle

A quantity of cocoa nib is ground after a heat pre-treatment. This is extracted in a soxhlet extraction apparatus using petroleum ether. The solvent is evaporated and the fat residue is weighed. The fat is dissolved in a mixture of ethanol and diethylether and the extracted free fatty acids are titrated with an ethanolic solution of potassium hydroxide.

5. Reagents

5.1 Petroleum ether, boiling range 40-60°C, dry and without evaporation residue (see note 11.1).

5.2 Mixture of ethanol and diethylether 1/1 (v/v/):

Mix 250 ml of 95% (v/v) ethanol with 250 ml diethylether. Neutralize the mixture just before use with the ethanolic potassium hydroxide solution (5.3) in the presence of 2.5 ml of indication solution (5.4).

5.3 Potassium hydroxide solution, 0.1 mole KOH (p.a.) in 1 L ethanol 95% (v/v) (Merck art. nr. 9115 or similar) and accurately standardized using potassium hydrogen phthalate (p.a.):

Weigh into a 250 ml Erlenmeyer flask approximately 400 mg of potassium hydrogen phtalate to the nearest 0.1 mg. Add a few drops of phenolphthalein and titrate with the ethanolic potassium hydroxide solution until the solution turns pink for at least 10 seconds (calculation see 9.1).

5.4 Indicator solution, dissolve 10 g of phenolphthalein in 1 L ethanol 95% (v/v) in a 1 L volumetric flask.

6a Materials: (see note 11.1)

6a.1 Defatted glass beads, diameter approximately 5 mm.
6a.2 Fat-free extraction thimbles, e.g. S&S no. 603 (33 * 94 mm).
6a.3 Fat-free round filters, diameter 15 cm, e.g. S7S no. 597.
6a.4 Defatted cotton wool.
6a.5 Pumice stones or other anti-bumping agents, fat free.

6b Apparatus: (see note 11.2)

6b.1 Soxhlet extractor, siphon capacity about 100 ml, NS 29 cone (male) at the bottom and NS 45 cone (female) at the top.
6b.2 Condensor, Dimroth or similar, with NS 45 cone and a drying tube or a small inverted beaker on top.
6b.3 Conical flask or flat bottomed flask, 250 ml with NS 29 cone.
6b.4 Heating apparatus for flasks, firesafe (sparkless).
6b.5 Vacuum drying oven set at 80°C, or a drying oven set at 103 ± 2°C.
6b.6 Dessicator, with drying agent at the bottom and with a tap in the lid.
6b.7 Analytical balance, 0.1 mg accuracy.
6b.8 Burette, 10 ml graduated in 0.02 ml.
6b.9 Suitable mill (p.e. Retsch, sieve 4 mm, level 1).
6b.10 (Prolabo) shaking machine according to the principle of Dangoumeau, with accessory 65 ml shaking containers and marbles. Every shaking container should be provided with an exactly fitting rubber ring between container and cover, to prevent leaking.
Marbles: every container contains 14 marbles: 2 marbles 15 mm diameter, 4 marbles 10 mm diameter and 8 marbles 6 mm diameter.
6b.11 Glass funnel, approx. 95 mm diameter at the top and a stem long enough to reach into the thimble. A metal gauze with openings (about 4 mm) is put in the funnel to prevent the marbles falling into the thimble.
6b.12 Microwave oven, 750 Watt, with turn-table.

7. Sampling

See International Standard: ISO/DIS 2292, "Cocoa Bean Sampling" - 1973, or similar procedure.

8. Procedure

8.1.1 Preparation of the test-sample:

Take about 250 grams of cocoa beans and remove the shells. Put the nib obtained in a 1 L beaker and place in the microwave oven. Heat for 3.5 minutes at 750 W, interrupted every 30 seconds in order to mix the nib (if the microwave oven has a lower maxium capacity, the time should be extended and multiplied by factor 750/ (maximum number of W). Cool down and grind the complete sample using a Retsch mill (sieve 4 mm), or a similar apparatus.

8.1.2 Analysis of the test material:

Weigh approx. 8 grams of the ground sample to the nearest 0.1 mg. Transfer the sample quantitatively to the shaking container, in which the marbles are already present. Add 30 ml petroleum ether, close the container and shake for 5 minutes in the shaking apparatus (6b.10).

Meanwhile place the soxhlet extractor on a conical flask or flat bottomed flask of 250 ml, containing some pumice stones. This flask and contents should be previously dried and weighed. (Ml [g]).
An extraction thimble in which some glass beads and a folded filter are put, is placed in the soxhlet extractor. Then the glass funnel with gauze is placed on the extractor.

After shaking the sample as above, pour the contents of the shaking container through the funnel into the thimble (marbles remain on gauze). Rinse the container and marbles with petroleum ether. Close the thimble with a wad of fatfree cotton wool. Add enough petroleum ether to the extractor to make it siphon twice. Attach the cooler to the soxhlet extractor, place the assembly on the heating apparatus and extract during four hours.

Disconnect the flask and evaporate the petroleum ether on the rotavapor. Dry the flask in the oven at 103°C in horizontal position. Then cool it down to room temperature in a dessicator for 30 minutes and weigh the conical flask and contents again (M2 [g]). Repeat the drying (for 1 hour). Weighing and cooling in the dessicator until the difference between two successive weighings does not exceed 2 mg.
Calculate the amount of fat which is obtained
(M [g] = M2 - M1).
Continue with the procedure to determine the FFA content (8.2).

8.2 FFA determination, titration

Take the flask with the fat obtained by procedure 8.1. Add 50 ml of the neutralized ethanol/diethylether mixture to the extracted fat. Titrate, while shaking, with the ethanolic solution of potassium hydroxide to the endpoint of this titration (phenolphtalein turns pink, lasting at least 10 seconds).
Note: If the quantity of 0.1 mole/1 potassium hydroxide solution required exceeds 20 ml, use 4 grams of sample instead of 8 grams.

9. Expression of results

9.1 Method of calculation and formula

F.F.A. content (acidity) is calculated by the formula:

F.F.A. = $\frac{282\ x\ V\ x\ C}{10\ x\ M}$ [% (m/m)] as oleic acid.

M = (M2 - M1)

In which:

282 = molecular mass of oleic acid
V = volume [ml] of the standardized potassium hydroxide used for the titration.
C = concentration [moles per litre] of the standardized potassium hydroxide solution.

$C = \frac{Wp}{Mp\ x\ Vp}$

Mp = molecular weight of potassium hydrogen phthalate
Vp = volume of potassium hydroxide solution
Wp = weight potassium hydrogen phthalate

M = mass [g] of the extracted fat.
M1 = mass [g] of the conical flask and pumice stones, before extraction.
M2 = mass [g] of the conical flask after extraction.

The result should be expressed with two decimals.

9.2 Repeatability

The absolute difference between two single test results obtained in repeatability conditions, shall not exceed the following values of r :

range = 0,5 - 2.0 ; r = 0.15 [% (m/m)]

9.3 Reproducibility

The absolute difference between two single test results, obtained in reproducibility conditions, shall not exceed the following value of R :

range = 0.5 - 2.0 ; R = 0.30 [% (m/m)]

10. Test report

The test report shall indicate the method used and the results obtained. It shall also mention any operating conditions not specified in the method or regarded as optional, as well as any circumstances that may have influenced the results.

The report shall include all details required for the complete identification of the sample.

11. Notes

11.1 Thimbles, filters and cotton wool must be checked for the absence of extractable matter: per analysis the total limit of the extraction-residue is 0.5 mg.
The petroleum ether must be free of evaporation residues: when the residue exceeds 1 mg per 150 ml, then the petroleum ether has to be distilled before use.

11.2 Round bottom flasks and water- or steam baths can also be used for the extraction, then however the extraction (boiling) is more difficult to control. Condensation of water in the condenser (high air humidity) should be avoided, it might wet the sample in the thimble and make the fat unextractable.

11.3 The round filter can be shaped like a bag, by folding it around a clean rod easily fitting into the thimble. This permits repeated use of the extraction thimble and also helps in preventing very fine cocoa particles being rinsed out of the thimble into the conical flask.

11.4 During extraction, the quantity of petroleum ether in the flask always has to be at least 50 ml.

12. Literature references

12.1 International Union of pure and Applied Chemistry (IUPAC). "Standard methods for analysis of oils fats and soaps", 6th Ed., section 2.201 (Butterworths, London).

12.2 International Standard ISO/DIS 729: "Oilseeds-Determination of acidity of oils" - 1988

12.3 Nederlands Normalisatie Instituut (NNI) : Ontwerp NEN 6332. "Onderzoekingsmethoden voor plantaardige en dierlijke olien en vetten. Bepaling van het zuurgehalte (zuurgraad zuurgetal).

12.4 Nederlands Normalisatie Instituut (NNI) : NEN 3103 "Chemische analyse, richtlijnen voor het stellen van de titer van titer vloeistoffen", hydroxide 1 en 3.

13. Collaborative study

16 Laboratories 6 samples each, in 3 split levels')
(without replicate)

') average values : 0.82 , 0.93 and 1.51 [% (m/m)].

12. METHODOLOGY FOR DETERMINATION OF MOISTURE CONTENT OF COCOA BEANS

Analytical Method 43

Determination of the Moisture Content of Cocoa Nib and Cocoa Beans

Introduction

This method describes the determination of the moisture content of cocoa nib or whole beans by drying and weighing.

1. Scope and Field of Application

Applicable to cocoa beans and cocoa nib prior to processing.

2. References

ICA method n°1 (1996) : "Determination of Moisture (Oven Method)"

3. Definition

The moisture content of cocoa nibs or beans is conventionally the loss in mass, determined by this method and expressed in [% m/m].

4. Principle

After grinding the sample is dried for 16 h in an oven, controlled at 103° C.

5. Apparatus

5.1 Suitable mill (p.e. Retsch, sieve 4 mm, level 1).
5.2 Ventilated oven, preferably fitted with a fan, capable of being controlled at 103 ± 2° C.
5.3 Dish with lid, made of metal resistant to attack under the conditions of the test, or of glass, with at least 35 cm^2 of useful surface (for example diameter 70 mm) and 20 to 25 mm deep.
5.4 Desiccator, containing an efficient desiccant.
5.5 Analytical balance.

6. Sampling and samples

See International Standard ISO/DIS 2292 - 1973, "Cocoa Beans Sampling",

or similar procedure.

7. Procedure

7.1 Preparation of the test sample:

From the test sample (normally 1 or 2 Kg of beans or nibs), weigh out approximately 250 g, then homogenise and grind it using a suitable mill, in order to give a coarse granulate. Make sure that the mill does not overheat, so as to prevent the moisture content from changing.

7.2 Determination:

Quickly transfer 10 g of the test sample into a previously weighed and dried empty dish (5.3) and lid. Cover the dish immediately with its lid and weigh to the nearest 0,1 mg.

Remove the lid and place the dish (5.3), containing the test portion, on its lid in the oven (4.2) controlled at 103 ± 2° C. Leave for 16 h ± 1 h, taking care not to open the oven door during this time. At the end of this period, remove the dish, cover it immediately with its lid and place it in the dessicator (4.4). Cool down to ambient temperature (approximatively 30 to 40 min) and weigh, still covered, to the nearest 0.1 mg.

Again put the sample in the oven for one hour, cool it down in the desiccator for 30 minutes and weigh. Should the difference between this weighing and the previous weighing exceed 0.1 %, this procedure should be repeated.

7.3 Number of determination:

Carry out two completely independent determinations, each on a quantity of nib or beans which has been treated individually: grinding, taking the test portion and drying.

9. Expression of results

9.1 Method of calculation and formula

The moisture content of the sample, expressed as a percentage by mass is equal to:

$$\frac{(A+B)-C}{B} \text{ x } 100 \text{ [\% (m/m)]}$$

In which:A = weight of dish + lid in g
B = weight of sample in g before drying
C = weight of dish + lid + sample in g after drying

9.2 Repeatability

The absolute difference between two single test results obtained in repeatability conditions, shall not exceed the following value of r :

r = 0.10 [% (m/m)] in the range : 3.5 - 5.0

9.3 Reproducibility

The absolute difference between two single test results obtained in reproducibility conditions, shall not exceed the following value of R :

R = 0.42 [% (m/m)] in the range : 3.5 - 5.0

10. Test report

The test report shall indicate the method used and the results obtained. It shall also mention any operating conditions not specified in the method or regarded as optional, as well as any circumstances that may have influenced the results.

The report shall include all details required for the complete identification of the sample.

11. Literature references

11.1 International Standard ISO 2291
Cocoa Beans - Determination of moisture content
second edition - 1980-12-01

12. Collaborative study

17 laboratories with 4 samples each : 2 uniform levels ')
in duplicate.
') average values : 3.82 and 4.57 [% (m/m)].

13. METHODOLOGY FOR DETECTION OF SPECIFIC OFF-FLAVOURS IN COCOA BEANS

Analytical Method 44

The Detection of Specific Off-Flavours
in Cocoa Beans

Introduction

The method allows the confirmation of the presence of specific off-flavours (e.g., smoke, mould and excessive acidity) in a sample of cocoa beans from a specific origin. The sensory testing procedure used is a modified form of the two from five test based on ISO 6658-1985.

1. Scope

The method is applicable to a sample of cocoa beans from a specified origin which is suspected of having a specific off-flavour.

The recommended number of assessors is 5 or 10. These assessors must be experienced in liquor tasting and have demonstrated ability in recognising the specific off-flavours of interest when they encounter them in cocoa liquor. However, assessors need not be highly trained.

2. Definitions

Smoke off-flavour - a flavour which is reminiscent of wood smoke, acrid smoke, burnt rubber, smoked bacon or soot.

Mould off-flavour - a flavour which is reminiscent of mouldiness, mustiness or dampness.

Hammy off-flavour - a flavour which is reminiscent of cured meats such as bacon or ham.

Acidic off-flavour - a flavour which is reminiscent of citrus fruits (lemon, lime, orange) or acetic acid (vinegar).

30% Sample - a composite sample taken from a parcel by sampling 30% of the bags as outlined in the Code of Practice of the International Cocoa Standard.

3. Principle

2 separate samples of cocoa liquor are prepared from a sample of cocoa beans suspected of having a specific off-flavour. These 2 liquors are

tasted amongst 3 other liquor samples prepared from three different samples of beans from the same origin and of the same type (e.g. fine or bulk) but which are known to be free of that specific off-flavour. These 5 liquors are assessed by a panel of 5 or 10 assessors.

Each assessor is presented with a set of the 5 liquors in a different random order and is asked to indicate the two samples in the set which are the strongest in the specified off-flavour. The probability of 3 assessors from a panel 5, or 4 assessors from a panel of 10, correctly identifying both suspect samples by chance, as having the strongest off-flavour, is near to 1 in 100.

4. Apparatus

4.1 Preparation of cocoa liquor

4.1.1 A fan assisted and ventilated oven capable of maintaining 148°C ± 2°C. The fan must stop automatically when the door is opened to avoid excessive cooling when introducing the beans to be roasted.

4.1.2 A 30cm x 30 cm roasting tray made of 0,5 cm x 0,5 cm wire mesh or other similar perforated metal tray capable of holding 330 g of beans in a single layer.

4.1.3 A motorised mortar and pestle mill with a porcelain set capable of grinding 300g of cocoa nibs while maintaining a temperature of 50°C ± 2°C (e.g. a Pascall Engineering Co. Ltd. model 1 "end runner" mill with 1kW mortar heater element, running at 120rpm).

4.1.4 A John Gordon & Co. bean breaker and winnower or scalpel and tweezers if bean shells are to be removed manually.

4.1.5 A pair of heat resistant gloves.

4.2 Sensory testing

4.2.1 A taste panel room which is quiet and odour free. Ideally, the room will be equipped with cubicles for 5 or 10 assessors, have red lighting and be maintained at close to 20 to 22°C (details of suitable room formats are described in The Official Journal of the European Community, L 248, Volume 34, 5th September 1991, pp 69-76). However, the minimum requirement is a room which is quiet and odour free and a suitable environment in which to make objective sensory measurements.

4.2.2 Bottled mineral water.

4.2.3 Disposable plastic spoons.

4.2.4 Spit cups.

4.2.5 15ml (or larger depending upon 4.2.6) opaque sample presentation vials with lids.

4.2.6 An aluminium heating block capable of holding 5 sample presentation vials at 60°C ± 5°C.

4.2.8 Sensory evaluation forms.

5. Procedure

5.1 Preparation of cocoa liquor

Preparation of cocoa liquor involves three processing stages. Roasting, beans, removing their shells and grinding the nibs into a liquor. The procedure described is based on the cocoa liquor preparation stages of ICA Analytical Method N°13/1971 "Manufacturing process of small scale chocolate samples" but has been amended to take into account the availability of more modern equipment.

Roasting the Beans

5.1.1 By a standard coning and quartering operation, a sub-sample of 330g cocoa beans is taken from the 30% Sample of cocoa beans.

5.1.2 Spread the 330g of beans onto the roasting tray to form a layer one bean thick.

5.1.3 The roasting oven (4.1.1) is set at 148°C and held at this temperature with the vent fully open for at least 15 minutes before the beans are roasted.

5.1.4 As quickly as possible to avoid excessive cooling of the oven, open the door and place the tray of beans in the middle of the top shelf and close the door. Roast the beans for exactly 30 minutes from the moment the oven door is closed.

5.1.5 Wearing the heat resistant gloves, remove the beans from the oven immediately the 30 minutes roasting period has elapsed.

Removing shells

5.1.6 If the John Gordon bean breaker & winnower is used, tip the beans into the bean breaker hopper and break them immediately after taking them out of the roasting oven.

5.1.7 Switch on the winnower and feed the broken beans slowly into the hopper. The winnower fan speed should be adjusted so that in the first pass of broken beans through the winnower, a shell free (less than 1% shell) nib fraction results.

5.1.8 Re-pass the shell fraction through the winnower a second time with the fan speed reduced so that a second shell free nib fraction is achieved. Repeat this step until no significant quantities of shell pass into the nib fraction.

5.1.9 Combine the shell free nib fractions.

5.1.10 If the shells are to be removed manually, use a scalpel to peel the shells from the beans.

Milling the Nibs to Produce a Liquor

The nibs can be milled immediately while still warm or if necessary, may be stored in a sealed food grade plastic container for not more than 24 hours.

5.1.11 Warm the mortar bowl of the mill (4.1.3) to 55°C.

5.1.12 With the mill operating in the grinding mode, slowly over a period of 2 to 3 minutes add the broken nibs into the bowl. Grind the nibs for 60 minutes at 55°C to produce a smooth liquor. During this period but particularly in the first few minutes of grinding, it will be necessary to remove material which builds up on the mortar scraper and add it back into the bowl. This can easily be done using a plastic spatula.

5.1.13 Remove the liquor from the bowl and from the pestle and collect it in a new food grade plastic sample pot of 250ml capacity. Place the lid on the pot.

5.1.14 Clean the bowl and pestle of the mill thoroughly, using disposable tissues, before using it to mill another sample.

Freshly prepared liquor samples can either be forwarded for sensory testing immediately or if required, may be stored for up to 12 months in a freezer at -10°C.

5.2 Sensory Testing

The sensory test is arranged to allow each assessor of a panel of 5 or 10 assessors to taste the liquors at 65°C in sets of 5, each set containing two presentations of the liquor samples suspected of having the specified off-flavour and 3 presentations of liquor samples which are known to be free from the specified off-flavour. Each set of 5 liquors to each assessor is arranged so that no two assessors receive the liquors and taste them in the same order.

Preparation of Cocoa Liquors for Sensory Testing

5.2.1 Code each of the sample pots containing the 5 liquors to be tasted with a different 3 digit random number.

5.2.2 Place the 5 liquors, in the coded sample pots, and 5 or 10 aluminium heating blocks (1 for each assessor) into an incubator set at 65°C to raise their temperature to 65°C. Liquors which have been stored in the freezer require to be incubated for 3 hours. No liquor should remain in the incubator for longer than 24 hours.

5.2.3 Code 5 opaque sample presentation vials per assessor, with the same 5 three digit numbers.

5.2.4 When all 5 coded liquors are at 65°C, thoroughly stir the liquor in each pot and fill each of the sample presentation vials with about 100ml of liquor from the sample pots coded with the same three digit random numbers. Place a lid on each vial.

5.2.5 Place the sample presentation vials into the aluminium heating blocks in sets of 5, according to the balanced presentation plan shown below, as viewed from in FRONT of the block, and return the aluminium heating blocks containing the sample presentation sets to the incubator set at 65°C.

For a panel of 5 assessors, assuming the 5 random 3 digit numbers are A, B, C, D and E, the sample presentation sets should be arranged as follows :

C,A,E,D,B E,C,B,A,D B,E,D,C,A A,D,C,B,E D,B,A,E,C

If 10 assessors are used, a further 5 presentation sets should be prepared as follows :

B,D,E,A,C D,A,B,C,E A,C,D,E,B E,B,C,D,A C,E,A,B,D

The liquors are now ready for presentation to the panel of assessors. They may be held in the incubators at 65°C for up to 4 hours during which time the sensory testing should be completed.

Conducting the Sensory Testing

5.2.6 Each assessor is be seated in a sensory testing cubicle equipped with 5 disposable plastic spatulas, a bottle of mineral water, a Cocoa Liquor Tasting Form, a pen and a spit spot.

5.2.7 Each assessor is presented with one aluminium heating block taken directly from the incubator set at 65°C and containing 5 liquors arranged in one of the sample presentation sets. The block is placed in front of the assessor so that the FRONT label on the block faces the assessor.

5.2.8 The assessor is asked to read and then carry out the instructions given on the Cocoa Liquor Tasting Form. An example of the Cocoa Liquor Tasting Form is shown below in section 5.2.9. The assessor is instructed that the samples must first be tasted in the order presented but may then be re-tasted in any order. Assessors must also be instructed not to communicate with each other during the test.

5.2.9 Cocoa Liquor Sensory Tasting Form

Taste the 5 coded cocoa liquor samples in the order presented from left to right and identify the two samples which exhibit the strongest off-flavour : (*specify the suspected off-flavour*)

The two samples are ------- and ------- (*Enter the appropriate 3 digit codes)*.

6. Expression of Results

Count the number of concurring responses. If the 2 samples of liquor prepared from the cocoa beans suspected of having the specific off-flavour are identified by 3 or more assessors of a panel of 5 assessors or by 4 or more assessors in a panel of 10 assessors, the off-flavour has been positively identified with greater than 95% confidence.

There is a small but finite probability that by chance the panel of assessors results will return a significant selection of a different pair of samples. In this event the quality of the three standard liquors should be questioned.

7. Test reports

In a two from five test the probability of a single assessor selecting the two correct samples by chance is 0.1 = p and the probability of an incorrect selection is 0.9 = q.

If two assessors carry out the test, the probabilities of all possible responses are calculated from the binomial expansion of $(p+q)^2=p^2+2pq+q^2$

thus the probability of 2 correct responses is p^2 = 0.01
the probability of 2 incorrect responses is q^2 = 0.81
the probability of 1 correct and 1 incorrect response is 2pq = 0.18
the probability of all possible responses is then $p^2+2pq+q^2=1.00$

And so for 5 assessors the probability of all possible responses is $(p+q)^5=p^5+5p^4q+10p^3q^2+10p^2q^3+5pq^4+q^5$

The probabilities are then :

all 5 correct responses	p^5	=0.00001
4 correct and 1 incorrect responses	$5p^4q$	=0.00045
3 correct and 2 incorrect responses	**$10p^3q^2$**	**=0.0081**
2 correct and 3 incorrect responses	$10p^2q^3$	=0.0729
1 correct and 4 incorrect responses	$5pq^4$	=0.32805
all 5 incorrect responses	p^5	=0.59049

In a similar way it may be calculated that the probability of 4 correct responses from 10 assessors is = **0.018** and the probability of 5 correct responses from 10 assessors is **0.0016.**

8. Literature references

The Official Journal of the European Communities, L248, Volume 34, 5th Sept. 1991, pp 69-76.